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DESIGN, CONSTRUCTION, AND INITIAL EXPERIMENTS WITH  
HIGH HYDROSTATIC PRESSURE EXTRUSION DEVICE

2248

BY

EDWARD MONIZ JORGE, 1944-

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A

THESIS

submitted to the faculty of  
UNIVERSITY OF MISSOURI - ROLLA  
in partial fulfillment of the requirements for the  
Degree of

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(advisor)

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K. L. Mayhew

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## ABSTRACT

The purpose of this study was to design and operate equipment with which the study of polymer extrusion would be possible at hydrostatic pressures up to 50,000 psi. By hydrostatic pressure is meant the pressure of the region of the extrudate. A study of the effects of back pressure on the extrusion of silicone gum was attempted.

## ACKNOWLEDGMENTS

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## I. INTRODUCTION

Since the 1940's the extrusion of polymers have been the object of investigation by various researchers. Pugh and Green (1) and Pugh (2) studied the effects on the extrusion of cold metals of pressure as high as 50 tons/in<sup>2</sup>. Their results in the tension tests showed that back hydrostatic pressure tended to reduce the tensile stresses in the neck. They also found a critical pressure at which brittle materials became ductile.

In this study an inquiry was made into the possibility of a correlation between extrusion of cold metals and of polymers at high hydrostatic pressure (the pressure in the region of the extrudate).



## II. REVIEW OF LITERATURE

When a material is subjected to a force it will respond to that force by undergoing a deformation or strain. Rheology is, by definition, the science of deformation and flow of matter. Different types of stress, such as, tensile, compressive and shearing are a result of the manner in which the stress is applied. The response to these different types of stress depends to some extent on different molecular mechanisms (3). Materials whose response is completely recoverable (return to original shape after stress is removed) are called elastic. Materials whose response is completely irrecoverable are called viscous. Materials which show both viscous and elastic response are called visco-elastic.

The rate of change of velocity transverse to the flow direction of a liquid is called the shear rate of the liquid. The shear force per unit area of the liquid (in a plane normal to the transverse direction taken above) is called the shear stress. These are the independent and dependent variables describing the steady-state rheology of the liquid; i.e. the shear stress acting on the liquid causes it to respond with a certain shear rate characteristic of the properties of that particular liquid.

For laminar flow of Newtonian liquids in capillaries under isothermal conditions, the flow may be described by the Hagen-Poiseuille equation. The ratio of the shear stress to shear rate, both evaluated at the wall, is called the viscosity of the fluid. The relationships of the variables may be written as:

$$\tau_w = (D \Delta P) / 4L \quad (1)$$

$$\gamma_w = 32Q / (\pi D^3) = 8U / D \quad (2)$$

$$\eta = \tau_w / \gamma_w \quad (3)$$

where:

$\tau_w$  = shear stress at the capillary wall,  $\text{lb}_f/\text{in}^2$ .

$D$  = diameter of capillary, in.

$\Delta P$  = pressure drop for flow through capillary, psi.

$L$  = length of capillary, in.

$\gamma_w$  = shear rate at the capillary wall,  $\text{sec}^{-1}$

$Q$  = volumetric rate of flow,  $\text{in}^3/\text{sec}$ .

$U$  = average velocity in capillary,  $\text{in.}/\text{sec}$ .

$\eta$  = viscosity,  $\text{lbs}_f \text{ sec.}/\text{in}^2$

Since Equations (1) and (2) were developed for laminar flow of Newtonian fluids they have to be modified to describe the capillary flow of non-Newtonian fluids. For Equation 1, a small end correction is considered effective to describe shear stress of non-Newtonian fluids. According to Dundevain and Klein (4) following Rabinowitsch (5) the true shear rate at the wall is obtained from:

$$\gamma_{tw} = \frac{1}{4} \left( 3 + \frac{1}{(d(\ln \gamma_w)/d(\ln \delta_w))} \right) \gamma_w$$

where  $\gamma_w$  is calculated by Equation 2.

Many different types of extrusion rheometers (6-10) have been used to study capillary flow of molten polymers and flow instabilities. In the 1940's Spencer and Dillion (11) and Nason (12) showed that there exists a critical shear rate at and above which the extrudate becomes deformed. Subsequently, the same effects were observed by Tordella (13) in the extrusion of polyethylene and other polymers.

Tordella found the origin of polymer deformation to be in the vicinity of the approach to the capillary. He found these distortions to be caused by a macroslip or tearing within the polymer as a result of slow relaxation times relative to the deformation rates - the stress imposed on the polymer exceeds its strength and fracture is the end result. The critical shear rate or that shear rate at which fracture occurs was found by Tordella (14) to be dependent on the polymer, its molecular weight, the extrusion temperature and the shape of the inlet to the capillary. He did not indicate how these variables affected the critical shear rate.

Bagley and Schreiber (15) using polyethylene with a carbon tracer found deformation of the extrudate to be caused by a break of the polymer just above the die inlet. With the oriented polymer at the center of the reservoir

breaking, it forced the surrounding polymer to be extruded; since the polymer surrounding the filament was not aligned, distortions resulted in the extrusion. By using a die with a large L/D ratio, Bagley and Schreiber found distortions of the extrudate minimal due to the fact that flow times in the capillary were much greater and the polymer had time to relax.

Tordella (14), Clegg (16), Bagley, Birks and Schrieber (15, 17), Bialas and White (18), Ballenger and White (19) made visual studies of the flow patterns of low density polyethylene using different types of dies. In the case of a flat entrance die, all of the above authors observed large dead spaces in the corners of the reservoir just above the capillary and that at the approach to the capillary the polymer moved through a funnel shaped region with the dead spaces defining its sides. It was also observed that the dead space to the right of the observer cycles in a counter-clockwise motion and that the left dead space cycles in a clockwise motion.

Ballenger and White (19) in studying the flow patterns in polymer melts above the capillary entrance found the dead regions to vary in size for different polymers. The amount of eddying occurring in the region above the capillary entrance, or the secondary circulating flows, were found to depend on the deviation of the melt from Newtonian fluid behavior. The relationship between

secondary flow patterns and rheological properties is being further investigated by them.

Clegg (16) in studying the extrusion of polymers postulated the theory that the shape of the extrudate not only depended on the shear stress but also on the rate of change of the shear stress at the capillary's entrance. Bagley and Schreiber (15), using high speed photography and black carbon as a tracer in polyethylene, studied the effects of die entry geometry on polymer melt fracture. Using dies with different conical angles, they concluded that melt fracture occurs at the same shear stress in both tapered and flat entry dies. Extrudate distortion was found by Bagley and Schreiber to be less severe in the case of a tapered die for two reasons. First in the case of a tapered die the dead region is either eliminated or reduced, thus diminishing the region of unoriented material. Second, the melt fracture lifetime was reduced. As mentioned previously, with the breaking and retracting of the highly oriented polymer, unoriented material surrounding it surges into the capillary entrance to take its place. This continues until the unoriented polymer suddenly breaks and retracts causing the highly oriented material to flow. The time it takes for the original material to reestablish itself is called the melt fracture lifetime and it decreases as the die entry angle decreases. As a consequence

of these two phenomena extrudate distortion is reduced.

Visual studies of various polymers made by Bialas and White (20) showed two distinct types of extrudate distortion. One type was characterized by screw thread appearance typical of high density polyethylene. The other type was characterized by an uneven swell of varying period appearance typical of low density polyethylene.

Matsuoka and Maxwell (21) in their study of the effects of extreme pressure on polymers found the structural changes, i.e. recrystallization and second-order transition of state, induced by addition of a high hydrostatic pressure (up to 70,000 atm) on linear high polymers to be time dependent. It was observed by them that the faster the material is compressed, the less pressure and deformation is required for the structural changes.

Bradbury and Leininger (22) studied the effect of pressures as high as 100,000 atms on various polymers. They found the density change of polyethylene to vary from  $-0.0001$  g/cc at a pressure of  $90 \times 10^3$  atms and a holding time of 2 min. to  $+0.0020$  g/cc at a pressure of  $98 \times 10^3$  atms and a holding time of 60 min. At least in the case of polyethylene they found the density change to be dependent on the holding time. It was observed by them that the changes in density did not reflect the changes in molecular composition. The physical changes

in polymers observed by Bradbury and Leininger at extreme pressures and elevated temperatures were small and no general compression effects were noticed.

Westover (23) and Maxwell and Jung (24) extruded polyethylene and other polymers under hydrostatic pressures and elevated temperatures were small and no general compression effects were noticed.

Westover (23) and Maxwell and Jung (24) extruded polyethylene and other polymers under hydrostatic pressures up to 25,000 psi. It was their observation that viscosity increased exponentially with pressure for most polymers used.

### III. EXPERIMENTAL

In choosing a polymer for this study certain requirements had to be met. The polymer had to have a molecular weight high enough to be able to sustain its shape after extrusion for a reasonable amount of time so that a photograph of the specimen could be taken and yet be ductile enough at room temperature to make its extrusion possible. Through trial and error it was found that a silicone gum produced by General Electric under the name of SE-30 with a molecular weight of about 600,000 and a viscosity of about 500,000 poises fulfilled most of the requirements.

A hydraulic fluid was used to transmit pressure from a ram to the polymer. The fluid used was a NATO H-515 fluid obtained from a surplus outlet with a label of OHA Hydraulic Fluid Petro. The fluid was a type of red oil with no high paraffins which would precipitate at high pressure.

#### A. Equipment

##### 1. Description of Rheometer

Two vessels capable of sustaining a pressure of 60,000 psi at 560°F were used in this experiment (see Figure 1). One of these vessels, which shall be referred



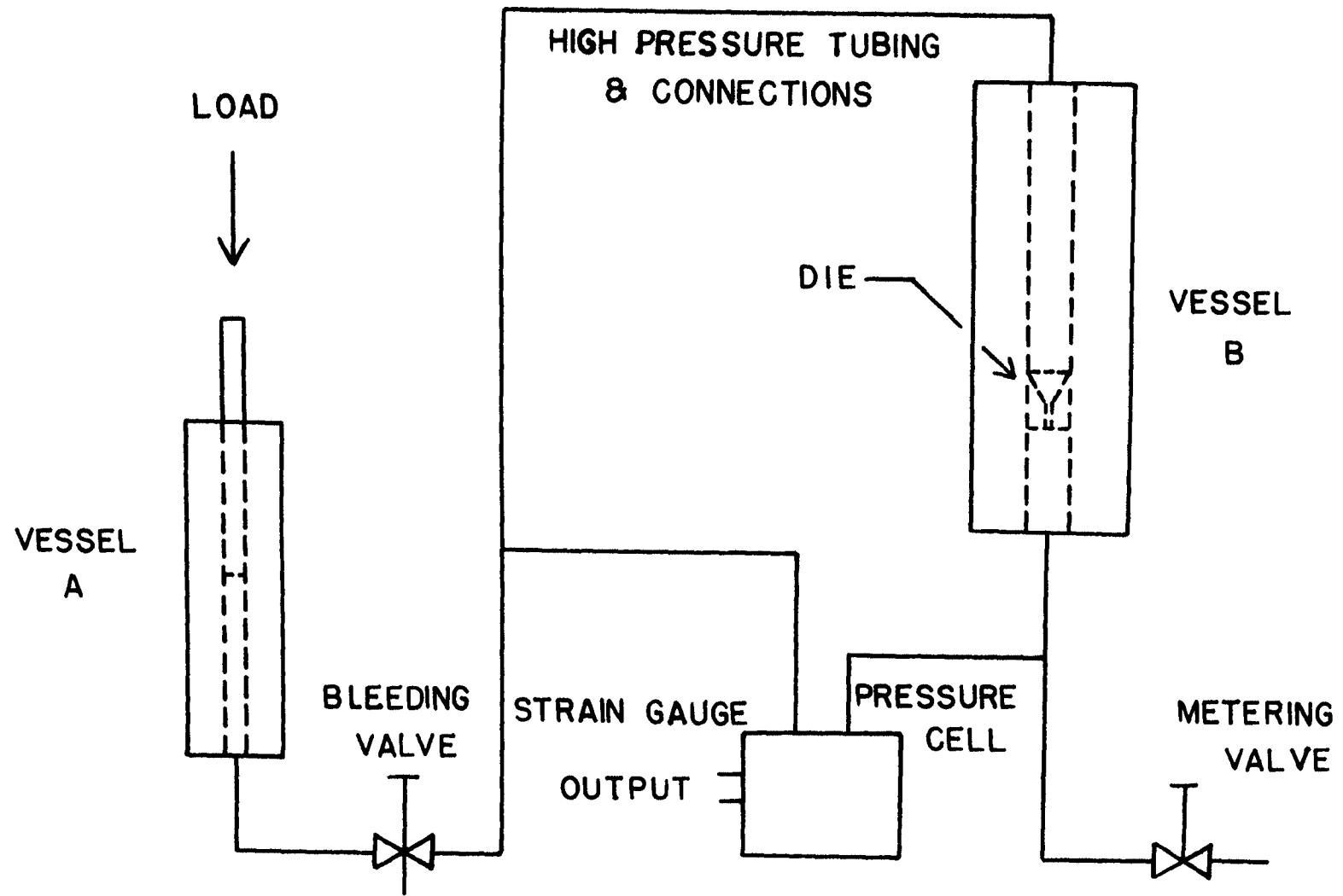


FIGURE 1. EXTRUDING APPARATUS

to as Vessel A, consisted of a one piece barrel with an I.D. of 0.804 in. and a piston made of hardened steel with a stroke of 6 in. Sealing between the piston and cylinder was attained by a vyton "O"-ring fitted near the inserting end of the piston.

Vessel A was mounted on a Riehle Testing Machine with a load capacity of 60,000 lbs and capable of driving at speeds as low as  $3.36 \times 10^{-2}$  in./hr. The Riehle Testing Machine, which was a floor model, was used as a rheometer.

The second high pressure vessel, which shall be referred to as vessel B, also consisted of a one piece barrel with an I.D. of 1 in. Vessel B was mounted on a platform which itself was mounted on a rolling table. This was done so that the equipment could be moved to and from the Riehle Testing Machine with less difficulty so that other investigators could use the testing machine.

Vessel B contained a sleeve 6 in. long which supported a capillary die. The die was made of carbon steel and had a conical entrance of  $60^{\circ}$ . It had an I.D. of 0.0043 in. and a L/D ratio of 5.81. A rubber "O" ring fitted around the die prevented any leakage between the die and the vessel wall. Sealing at the top and bottom of vessel B was accomplished by two Teflon "O" rings.

High pressure stainless steel tubing with an O.D. of  $\frac{1}{4}$  in. connected vessel A to vessel B. A metering valve with the capacity of sustaining pressures of 60,000 psi was connected by high pressure tubing to the base of vessel B.

## 2. Description of Differential Pressure Cell

In order to measure the pressure drop across the die, a device using a strain gauge was used. The differential pressure cell (Fig. 2), cylindrically shaped with an outside diameter of  $4 \frac{1}{2}$  in., was made of stainless steel and composed of two sections.

One of the sections, 2 in. long, contained two small holes  $\frac{1}{2}$  in. apart along its longitudinal axis through which two electrodes were inserted. Figure 3 shows a sketch of the electrodes. A plastic sleeve surrounded the electrodes serving as an insulator and at the same time preventing any leakage around them.

The other section,  $3 \frac{5}{8}$  in. long, contained a hole  $1 \frac{5}{8}$  in. long made up of two parts. The first  $1 \frac{3}{8}$  in. constituted the first part with a diameter of 1 in.; the other  $\frac{1}{4}$  in. constituted the second part with a diameter of  $\frac{3}{4}$  in. Each part of the cavity had a  $\frac{1}{8}$  in. diameter hole connecting it to a standard high pressure tubing connection machined on the outer surface of the pressure cell.

A  $\frac{1}{16}$  in. thick diaphragm made of stainless steel

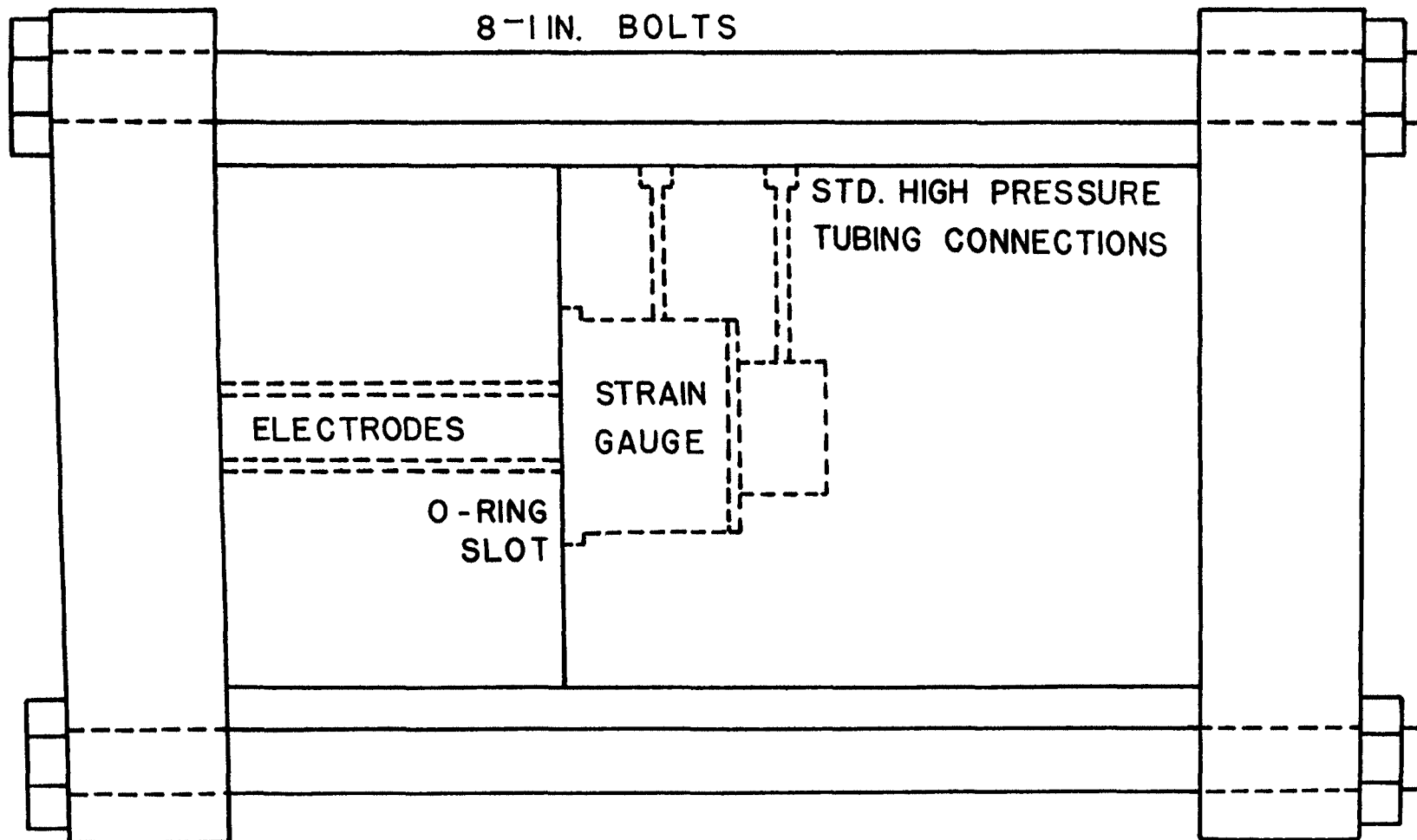


FIGURE 2. DIFFERENTIAL PRESSURE CELL

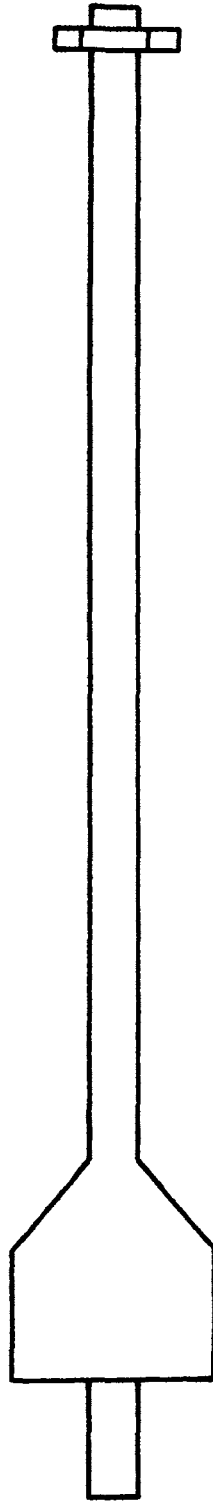


FIGURE 3. ELECTRODE

separated the two parts of the cavity. A strain gauge with a resistance of 120 ohms was glued to the surface of the diaphragm. The strain gauge leads were connected to the electrodes so that a bridge (Baldwin - Lima - Hamilton Sr-4) would be used to measure strain. The diaphragm was held in place by a hollow screw with an O.D. of 1 in. and sealing between the two parts was accomplished by two Teflon washers.

A rubber "O" ring fitted around the 1 in. hole sealed against leakage between the two pressure cell's sections which held together by two 2 in. thick plates containing 8 - 1 in. carbon steel bolts in a 5 5/8 in. circle. One of these plates had drilled through its center a 1 1/2 in. hole for the electrical connections of the strain gauge.

## B. Experimental Procedure

### 1. Differential Pressure Cell

In order to convert strain in in./in. to pressure in psi, the differential pressure cell was calibrated. After the strain gauge had been glued on the diaphragm, its lead wires soldered to the electrodes, the strain gauge and lead wires covered with waterproofing, and the bolts encircling the pressure cell tightened, both cell, one on each side of the diaphragm, were filled

with hydraulic fluid. Since the holes leading to these cells were only  $1/8$  in. in diameter, a syringe with a  $2\frac{1}{2}$  in. long needle was used to fill the cells with fluid, thus preventing any air from being trapped inside the pressure cell.

To cause the seating of the diaphragm to be better as the pressure differential increased, it was decided to have the higher pressure on the cavity with the larger diameter thus forcing the strain gauge to indicate compression instead of tension. This had no particular effect other than make the strain gauge a little less sensitive.

The standard high pressure connection of the larger diameter cell was connected to vessel A and to a pressure gauge which read up to 1000 psi. The strain gauge tended to drift somewhat when first used. In order to eliminate the strain gauge drift, it was pressurized to 1000 psi several times until the strain gauge gave consistent readings.

A Baldwin - Lima - Hamilton SR-4 strain indicator was used to read strains. By controlling the rate at which the Riehle Testing Machine was being driven, desired pressures were attained for a pressure vs. strain calibration curve. The pressure cell was calibrated from 0 - 1000 psi since the pressure drop across the die occurred in this range.

After the pressure cell had been calibrated, high pressure tubing was connected from the other cell to the base of vessel B thus giving a direct measure of the pressure drop across the die.

## 2. Rheometer

The polymer was loaded in vessel B directly above the die. Quick loading was found to be the best way of preventing any air from being trapped in the polymer. A small piston with a rubber "O" ring seal was placed directly above the polymer to drive it evenly. After the polymer had been loaded and the driving piston placed in its proper position, the sections below the die and above the driving piston were filled with hydraulic fluid. Since vessel B weighed in excess of 100 lbs, a hoist was designed to make handling of vessel B easier.

To fill the section below the die with hydraulic fluid, the vessel had to be inverted to prevent air from being trapped. After vessel B had been filled at both ends with fluid it was ready to be connected to the other pieces of equipment. After vessel A and all of the connecting tubing had been filled with hydraulic fluid by use of a hand pump and all of the connections joined together, the equipment was ready for use.

With the metering valve slightly open, the rheometer was activated. By controlling the rate at which the piston in vessel A was displaced and also by controlling the



setting on the metering valve, the desired pressure and rate of extrusion were attained. The rate of polymer extrusion was measured by measuring the rate of hydraulic fluid displaced by the extrudate.

After equilibrium had been reached, the load on the rheometer was read, the extrusion rate measured by finding with a stop watch the time required to extrude 0.2 cc of polymer and the pressure drop across the die was read on the strain indicator.

As soon as each experiment was completed, the bottom of vessel B was disconnected and the extrudate removed. A photograph of the polymer extruded was taken as a permanent record.

#### IV. DISCUSSION

##### A. Equipment Development

In developing the equipment for the study of polymer extrusion at high hydrostatic pressure the biggest problems arose in the design of the differential pressure cell and in the system controlling the flow rate.

Since, a pressure gauge able to measure the pressure drop across the die at pressures as high as 50,000 psi was very expensive, a different method had to be found to measure it. It was finally decided that a differential pressure cell containing a diaphragm upon which a strain gauge was mounted would perform satisfactorily.

In the design of the pressure cell, the biggest concern outside of safety was accuracy. The diaphragm not only had to be thick enough to prevent its deformation from exceeding the elastic limit, but it also had to be thin enough to give an easily measurable deflection. The equations used in the design of the diaphragm were (25):

$$f_{\max} = (3P/16)(d/t)^2 \quad (5)$$

$$f_{\text{at center}} = ((1+\mu)d^2)/(64(t^2/6))P \quad (6)$$

$$y_{\max} = Pd^4/1024D \quad (7)$$

where:

$f$  = stress, psi.

$P$  = pressure, psi.

$d$  = diameter of diaphragm, in.

$t$  = thickness of diaphragm, in.

$\mu$  = Poisson's ratio

$y$  = deflection of diaphragm, in.

$D$  = flexural rigidity =  $Et^3/12(1-\mu^2)$

$E$  = modulus of elasticity

The whole pressure cell including the diaphragm was made of stainless steel to prevent corrosion. Since the properties of the stainless steel were unknown, it was assumed that its modulus of elasticity was  $30 \times 10^6$  psi and its modulus of rigidity  $12 \times 10^6$  psi.

In securing the strain gauge, which had a resistance of 120 ohms, to the diaphragm, Eastman's glue was used. In order to prevent the hydraulic fluid from coming into contact with the strain gauge a waterproofing material was spread on its surface. No matter how many layers of waterproofing were used, however, it was impossible to prevent the hydraulic fluid from gradually changing the strain gauge's response, a fact that went unnoticed until all of the experimental data had been collected.

To solve this problem, it is proposed that the pressure cell be filled with mercury instead of hydraulic fluid. With the strain gauge and its leads covered with waterproofing, mercury could be used without any detrimental effects. Because of mercury's high density, there is no problem in ever having the hydraulic fluid from coming into contact with the strain gauge.

In sealing the two sections composing the pressure

cell, a C-type ring was first used. Because of the poor results obtained a Viton "O" ring was later substituted with excellent results.

The control of very small flow rates at high pressures was almost impossible with available high pressure metering valves due to the tremendous pressure drop across them. These valves were very erratic in their flow rate control behavior. At a given valve setting the flow rate tended to decrease until it was non-existent even though the valve seemed to be clean and the differential pressure across the valve was increasing.

It is proposed that a completely new flow control system, one not using any type of valve, be used. Since after a certain pressure has been attained the rheometer extrudes at a constant pressure (by fixing the back hydrostatic pressure), a constant flow rate should be attained.

Using the principle of a dead load tester, a new small diameter, high pressure vessel (Fig. 4) containing a piston with a platform on top for loading weights will give a constant back pressure. By using the correct load, the back pressure can be maintained at a constant desired pressure. An "O" ring on the piston should be used to seal it against the cylinder wall. This system would be filled with hydraulic fluid and attached to the bottom of vessel B. The extrusion of the polymer will

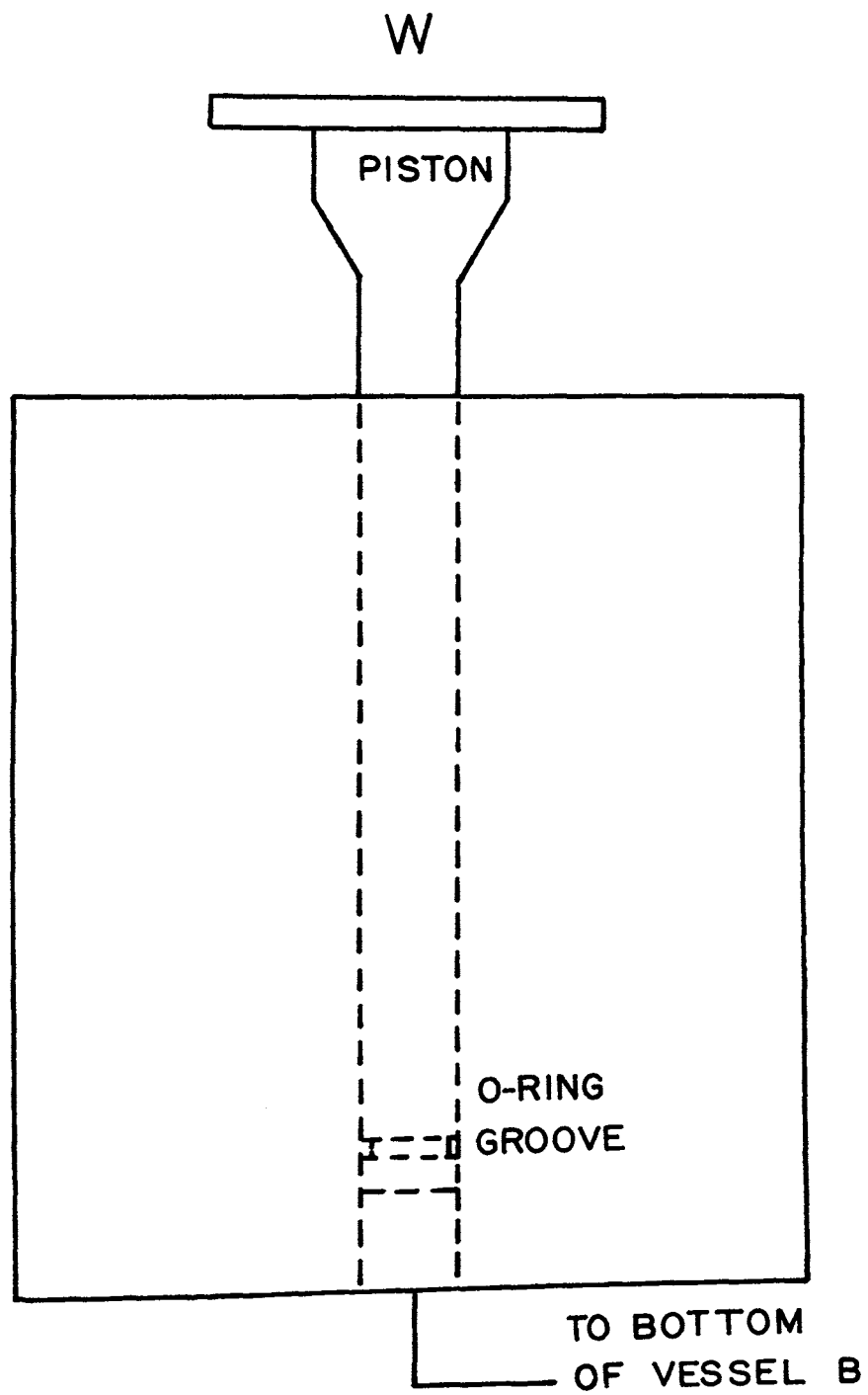


FIGURE 4. FLOW RATE CONTROL VESSEL

cause the hydraulic fluid at the bottom of vessel B to be displaced which in turn will cause the piston of the new vessel to be pushed up. The rate at which the piston in the new vessel moves upward will give a means of more accurately measuring the flow rate of the extrudate.

Because quite a bit of polymer had to be extruded before equilibrium was reached, the extrudate many times tended to get compacted in the lower section of vessel B so as to lose its shape. This caused wasted time in reperforming experiments. A direct line containing a valve from vessel A to the bottom of vessel B should be used. During the loading process (pressure increase) the upper and lower sections of vessel B will have identical pressures. When the desired hydrostatic pressure is attained, the valve should be turned off and the rate of extrusion adjusted by adjusting the rate at which the rheometer is moving thus fixing the pressure on the upper section of vessel B.

Alignment of vessel A with the rheometer must be done with extreme care. Due in part to poor alignment and to poor craftsmanship in the machining of vessel A, its piston galled the side of the cylinder when first used. In designing vessel A, the hardness of the piston and cylinder must not be the same, yet the piston and cylinder are both made of very hard materials. A piece

of plywood or cardboard was placed under vessel A to create a self-aligning system in case the vessel was not initially perfectly aligned.

Another problem encountered was that of the hydraulic fluid channeling through the polymer. The problem was solved by the design of a small piston to push the polymer into the die. An "O" ring on the piston prevented any hydraulic fluid from being mixed with the polymer.

The problem of the oil softening the extrudate was never satisfactorily solved. It is the opinion of this investigator that the softening was caused by the hydraulic fluid used to collect the extrudate in the section below the die in vessel B, since no such effects were observed in the extrusion of the polymer in air. This softening effect tended to minimize extrudate distortions, a fact which should be taken into consideration. Harder polymers were sought in order to alleviate this problem, but the General Electric SE-30 silicone gum is the best polymer used so far.

Taking a good picture of the extrudate sample was difficult because the polymer was transparent. The extrudate sample was placed on a tracing table to be photographed using a Polaroid camera with the shutter speed and f - stop adjusted to get the best contrast. Best results were obtained when the oil-wet extrudate was on a piece of tissue paper, through which the light shone.

## B. Experimental Results

Due to the problems mentioned in the previous section, it was with extreme difficulty that data was obtained up to a pressure of 4930 psi.

Extrusion without back pressure of the silicone gum was done between a flow rate of 0.0312 cc/min. which occurred at a pressure of 128 psi and a flow rate of 0.918 cc/min. which occurred at a pressure of 355 psi. It should be noted that these pressures represent the pressure drop across the die. It was not possible with the equipment used to attain flow rates small enough to obtain a perfectly smooth extrudate although at a flow rate of 0.0312 cc/min. only minute ridges were visible. In the range of the flow rates mentioned above, the distortion of the extrudate went from minute ridges to screw thread like distortion at a flow rate of 0.177 cc/min. and finally to a sausage like appearance at a flow rate of 0.918 cc/min. (see Figs. 5,6,7).

Figs.(8,9,10) show the polymer extruded under high hydrostatic pressures. The extrudate's diameter was over twice as large as the 0.043 in. diameter of the die; die swell was quite noticeable. Although the die swell seems to be dependent on fluid flow rate (the lower the flow rate the more noticeable die swell) no definit conclusions can be drawn since there was not enough data and the data taken was not consistent enough.





FIGURE 5. POLYMER EXTRUDED AT A PRESSURE OF 128 PSI. AND A FLOW RATE OF 0.0312 CC/MIN.

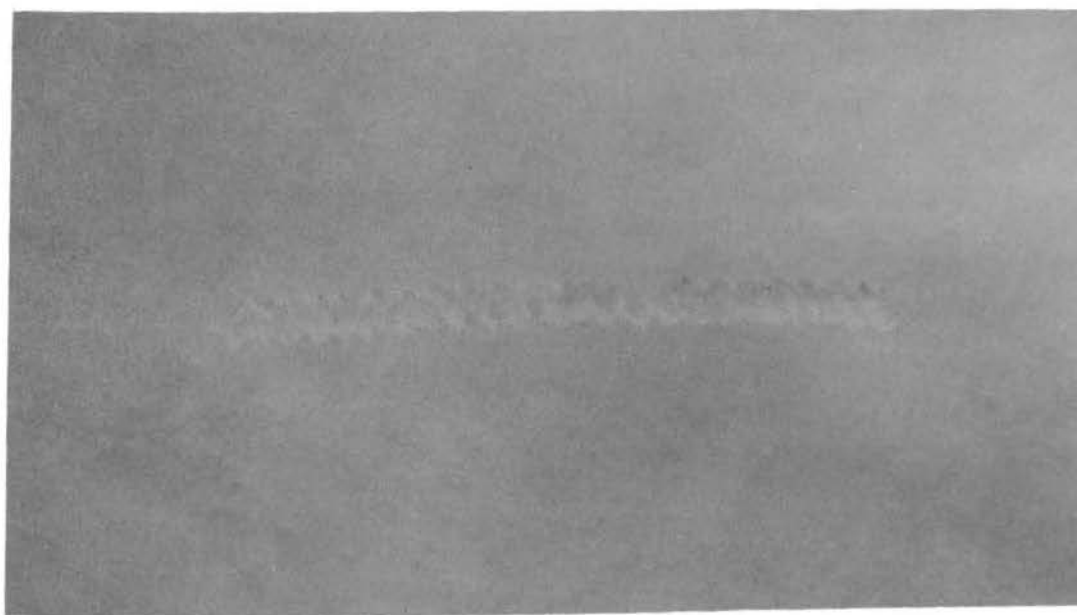


FIGURE 6. POLYMER EXTRUDED AT A PRESSURE OF 290 PSI. AND A FLOW RATE OF 0.434 CC/MIN.

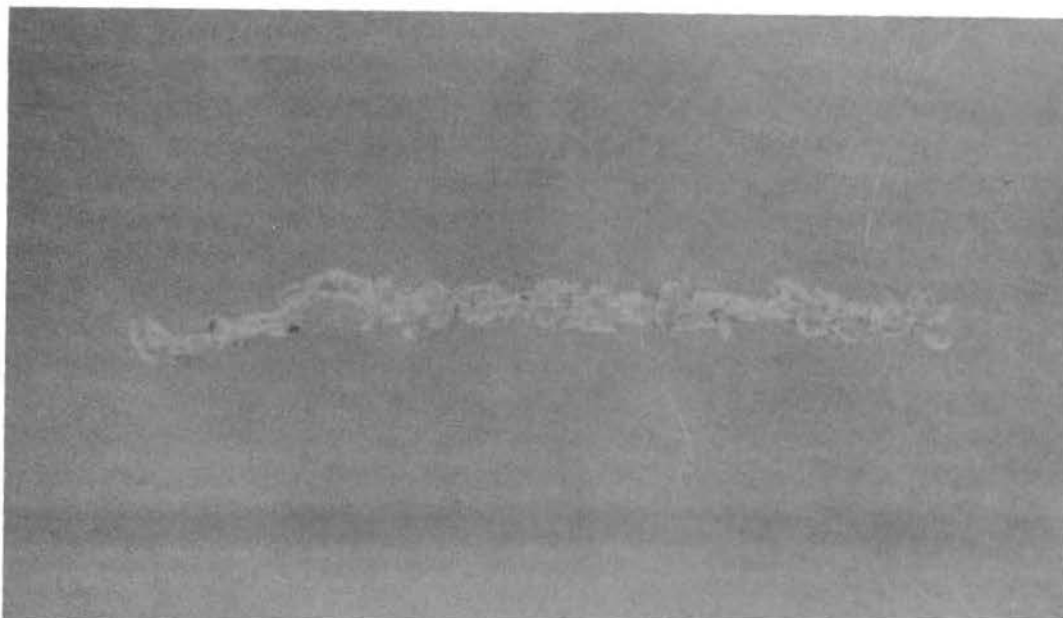


FIGURE 7. POLYMER EXTRUDED AT A PRESSURE OF 356 PSI. AND A FLOW RATE OF 0.918 CC/MIN.

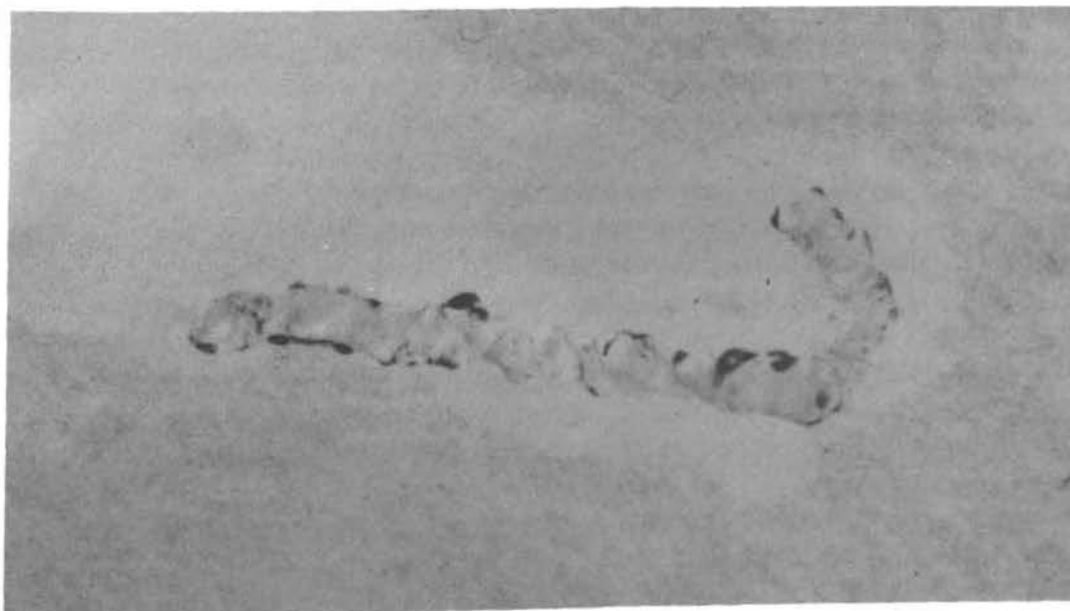


FIGURE 8. POLYMER EXTRUDED AT A PRESSURE OF 591 PSI. AND A FLOW RATE OF 0.444 CC/MIN.

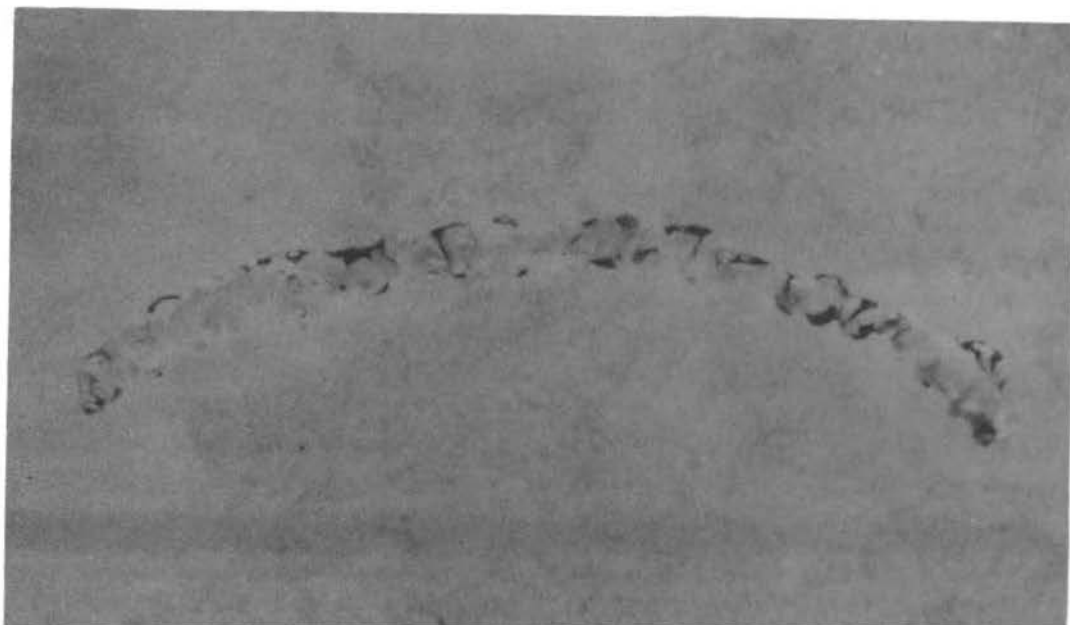


FIGURE 9. POLYMER EXTRUDED AT A PRESSURE OF 2370 PSI. AND A FLOW RATE OF 0.444 CC/MIN.

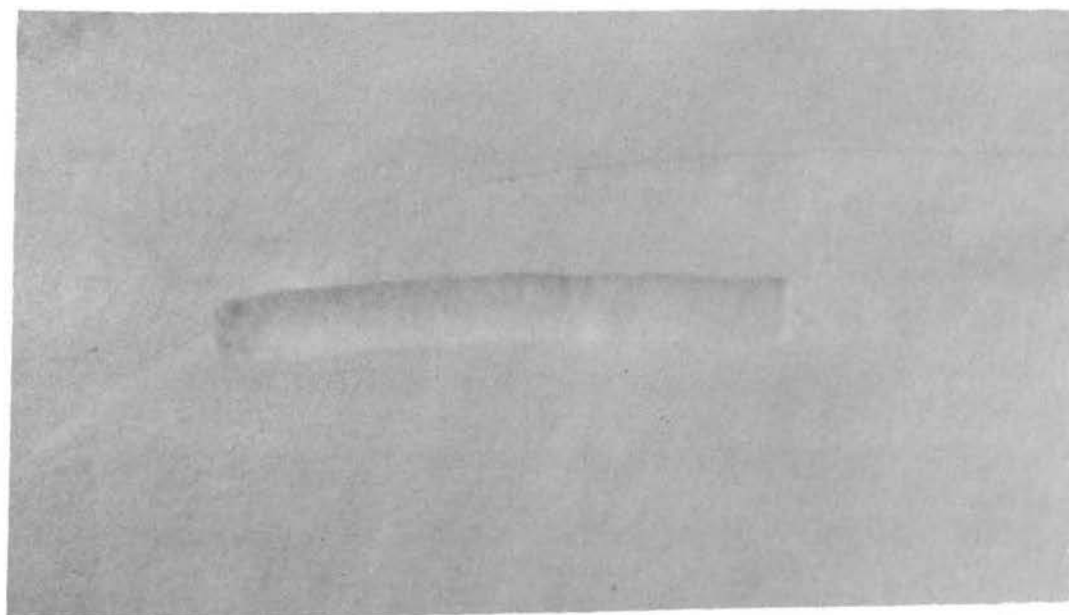


FIGURE 10. POLYMER EXTRUDED AT A PRESSURE OF 2370 PSI. AND A FLOW RATE OF 0.134 CC/MIN.

Fig. (11) is a plot of die differential pressure (and wall shear stress) versus flow rate (and shear rate) without back pressure. The differential pressure for calculating wall shear stress was taken to be the pressure on the piston in vessel A. Wall shear rate was assumed to be approximately  $8U/D$ . This calculation neglects any elastic entrance and exit effects on differential pressure and assumes no plug flow, which is not necessarily true. This figure shows that the polymer was a non-Newtonian fluid—an expected result.

Because the hydraulic fluid tended to gradually detach the strain gauge from the diaphragm, a condition that was not noticed until the experimental part of this study had been completed, the differential pressures across the die measured by the pressure cell will not be presented. Due to this fact, increased viscosity at high hydrostatic pressure observed by others could not be confirmed.

At a hydrostatic pressure of 1000 psi. it was observed that the piston in vessel A had a 7 psi drag stress. Since this amounted to less than 1% of the total force, it was neglected.

In the extrusion of silicone gum, with a hydrostatic pressure, no melt fracture effects of pressure were noticed by this observer. In all the runs using hydrostatic pressure, which ranged from about 450 psi to about 4700 psi, a distinct melt fracture was noticed at approximately the

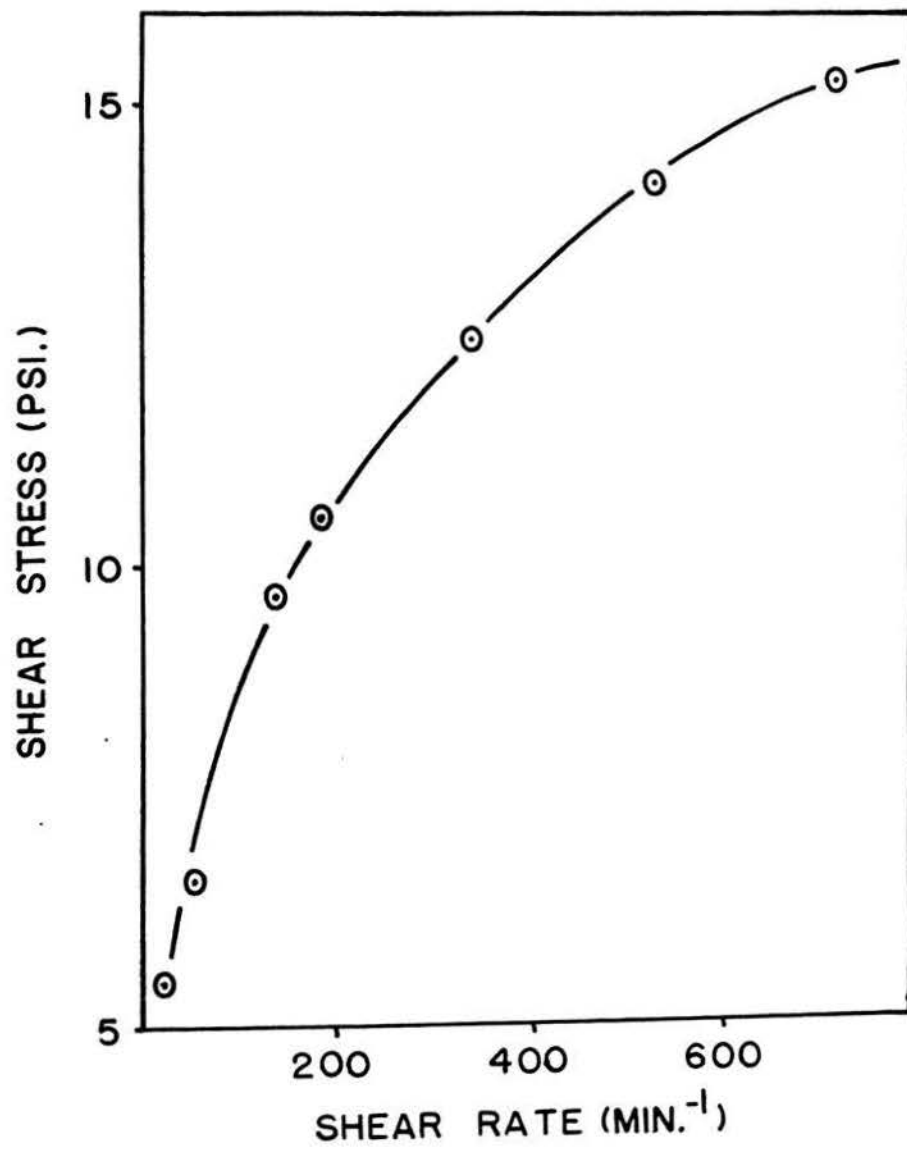


FIGURE 11. SHEAR RATE VS. SHEAR STRESS WITHOUT HYDROSTATIC PRESSURE

same flow rate (approximately 0.13 cc/min. or a shear stress of 8.6 psi ).

Following is a list of pressures and flow rates used in this experiment for the extrusion of silicone gum SE-30.

TABLE I

Results of the extrusion of SE-30 without hydrostatic pressure:

Run	Flow Rate (cc/min.)	Velocity (cm/min.)	Shear Rate (min. <sup>-1</sup> )	Shear Stress (psi.)	P (psi.)	Die Swell Ratio
1	0.0312	0.334	24.5	5.50	128	2.4
2	0.0764	0.815	59.8	6.60	153	2.1
3	0.177	1.89	139	9.72	226	screw
4	0.236	2.52	185	10.58	246	screw
5	0.434	4.64	340	12.46	290	screw
6	0.679	7.25	531	14.14	329	screw
7	0.918	9.80	720	15.30	356	sausage

TABLE II

Results of the extrusion of SE-30 using hydrostatic pressure:

Run	P (psi.)	Flow Rate (cc/min.)	Velocity (cm/min.)	Shear Rate (min. <sup>-1</sup> )	Die Swell Ratio
1	591	0.444	4.75	348	screw
2	591	0.124	1.32	97	2.9
3	591	0.017	0.18	14	2.8
4	1185	0.500	5.34	394	screw
5	1185	0.116	1.24	92	2.5
6	1185	0.064	0.69	51	3.2
7	2370	0.444	4.75	348	screw
8	2370	0.134	1.42	102	3.0
9	2370	0.063	0.67	50	3.1
10	4930	0.119	1.27	92	2.5
11	4930	0.444	4.75	348	screw



## V. CONCLUSION

The following conclusions can be drawn from this study:

- 1) It is possible to measure the pressure drop across the die using a strain gauge transducer. With selection of the correct strain gauge, and elimination of the separation problem, accuracy is excellent.
- 2) Control of small flow rates at high hydrostatic pressures is impossible by the use of high pressure metering valves, therefore the proposed new vessel for pressure control is necessary for further work.
- 3) Although die swell was observed, not enough consistent data was obtained to find its cause.
- 4) Hydrostatic pressure as high as 4930 psi was found to have no effect on the melt fracture of silicone gum.

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## VII. VITA

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